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(54) Detergent composition in tablet form.

(57) A tablet of compressed particulate detergent composition suitable for washing fabrics in a washing machine comprises a detergent-active compound, a detergency builder, a bleach system comprising sodium percarbonate, and optionally other detergent ingredients. The percarbonate is separated from any ingredient of the composition detrimental to its stability by segregation in a discrete region of the tablet, for example, a separate layer, optionally together with a compatible inorganic salt, for example, sodium carbonate, as diluent. Advantageously the diluent is spray-dried and also contains a polymeric binder.

EP 0 481 793 A1

TECHNICAL FIELD

The present invention relates to detergent compositions in the form of tablets of compacted detergent powder containing sodium percarbonate.

BACKGROUND AND PRIOR ART

Detergent compositions in tablet form have been known for many years although the form has never achieved great popularity on the market. In principle, tablets offer several advantages over powder products: they do not require measuring and are thus easier to handle and dispense into the the washload, and they are more compact, hence facilitating more economical packaging and storage.

One difficulty that has been experienced in the formulation of detergent tablets is the incorporation of bleaching ingredients, especially when the presence of bleach-sensitive ingredients such as enzymes is also desired: in a compressed tablet, the ingredients are much more intimately associated with one another than in a powder, and any adverse interactions and instability will be exacerbated.

GB 911 204 (Unilever) discloses layered detergent tablets containing persalt bleach, for example, sodium perborate, and certain bleach activators, for example, sodium acetoxybenzene sulphonate and phthalic anhydride. To avoid destabilisation, the bleach activator is segregated from the remaining tablet ingredients, including the persalt bleach, in a separate section or layer.

In contrast, EP 395 333A (Unilever) discloses a detergent tablet containing sodium perborate in conjunction with one or more bleach-sensitive ingredients - tetraacetylethylenediamine or similar bleach activator, enzyme, fluorescer, or any combination of these - as well as detergent-active compounds, detergency builders and optionally other ingredients. The persalt is not segregated from the bleach-sensitive ingredients but, surprisingly, the tablet is stable with no more loss of bleach, enzyme or fluorescer performance on storage than in a powder of the same composition.

Sodium percarbonate, $Na_2CO_3.1.5H_2O_2$, is less stable than sodium perborate in the presence of moisture: unlike sodium perborate, which exists as a tetrahydrate and a monohydrate, it is anhydrous, and when it absorbs water hydrogen peroxide is liberated and this decomposes readily. Stabilisation of sodium percarbonate in detergent powders has long been recognised as a difficulty: its incorporation in tablets would therefore be expected to be even more problematic.

The problem becomes especially acute if sodium percarbonate is to be included in a detergent powder with a high free moisture content, when it tends to become deactivated on storage. This situation applies in particular to powders containing crystalline alkali metal aluminosilicates (zeolites), because those materials contain a large amount (about 10-15 wt% in zeolite 4A, for example) of relatively mobile water.

Detergent tablets containing sodium percarbonate are disclosed in US 3 953 350 (Kao) but the detergency builder is sodium tripolyphosphate. JP 60 015 500A (Lion) discloses tablets containing anionic surfactant, zeolite, sodium sulphate, and a sodium bisulphate/sodium percarbonate bleach system.

GB 1 505 274 (Colgate-Palmolive) discloses detergent compositions in the form of a plurality of small dosage units, for example sachets but preferably and specifically tablets, containing different ingredients that can be dosed individually by the consumer. A three-tablet system is described (Example 6) consisting of a detergent tablet (surfactant, builders, fluorescer, colourant), a separate builder tablet (additional builder), and a separate bleach tablet (sodium percarbonate and nonionic surfactant).

The present invention now presents a solution to the problem of how to prepare a storage-stable detergent tablet containing both sodium percarbonate and ingredients to which it is sensitive. Surprisingly, the tablet of the invention also shows improved dissolution in the wash liquor.

DEFINITION OF THE INVENTION

The present invention provides a tablet of compressed particulate detergent composition comprising a detergent-active compound, a detergency builder, a bleach system comprising sodium percarbonate, and optionally other detergent ingredients, wherein the percarbonate is separated from any ingredient of the composition detrimental to its stability by segregation in a discrete region of the tablet.

DETAILED DESCRIPTION OF THE INVENTION

Detergent tablets of the invention solve the problem of percarbonate instability by segregating that material and isolating it from other ingredients which may interact adversely with it. Greatly improved storage stability is observed even when the detergent base composition contains zeolite, with its large amounts of associated

mobile water. Surprisingly, the segregated tablets of the invention also dissolve more quickly in the wash liquor than do similar tablets in which the sodium percarbonate is simply mixed homogeneously with other ingredients before tabletting.

Percarbonate segregation

In the tablet of the invention, at least one discrete region comprising sodium percarbonate and optionally other ingredients compatible with sodium percarbonate is present. Other components such as detergent-active compound, detergency builder and any other ingredients of doubtful compatibility with sodium carbonate are excluded from the discrete region(s) in which the sodium percarbonate is segregated.

Various means may be used to achieve the segregation. The percarbonate may, for example, be isolated in a layer, core or insert, while other ingredients are present in other layers, or in the main body or matrix of the tablet. More than one layer, core or insert may be present. Another possibility is the use of relatively large granules or noodles distributed throughout the main body or matrix of the tablet, the granules or noodles being protected by coating or encapsulation. Suitable coating materials or encapsulants will readily suggest themselves to the skilled detergent formulator.

A preferred embodiment of the invention which is simple in structure and simple to manufacture is a tablet consisting of two layers: the first layer containing the percarbonate, and the second layer containing other ingredients. Each layer is preferably substantially homogeneous, that is to say, is the compaction product of a single particulate composition, although that particulate composition may have been prepared by mixing a number of components and all its particles will not necessarily be identical.

The percarbonate may be segregated alone, or together with one or more other ingredients that are fully compatible with it. It is generally preferred that a major proportion of the non-percarbonate ingredients should be separated from the percarbonate.

However, the stability of the percarbonate may actually be increased by segregating it together with a diluent in the form of a compatible inorganic salt. The salt is preferably in a finely divided or highly porous form having a preferred surface area, as measured by nitrogen absorption of 5.15m^2/g. It is believed that the inorganic salt contributes to percarbonate stability by acting as a moisture sink. One especially preferred inorganic salt is sodium carbonate, which of course also plays a useful role in the detergent composition as a whole, as a detergency builder and provider of alkalinity. It is believed that sodium carbonate may also contribute to percarbonate stability by reabsorption of any liberated hydrogen peroxide.

According to one especially preferred embodiment of the invention, the diluent is in the form of a spray-dried composition comprising the compatible inorganic salt, more preferably sodium carbonate, and a polymeric binder.

The binder must itself be stable to oxidation.
Preferred binders are acrylic and/or maleic polymers, for example, the acrylic/maleic copolymer sold commercially as Sokalan (Trade Mark) CP5 ex BASF. As well as their binder function which improves tablet integrity and allows tabletting without having to wet the percarbonate to any significant degree, polycarboxylate polymers of this type also have a useful detergency building and antiredeposition action.

In this embodiment of the invention, the discrete tablet region or layer is the compaction product of a particulate composition prepared by mixing sodium percarbonate with the spray-dried salt/polymeric binder granules. This particulate starting composition suitably contains from 30 to 70 wt% of sodium percarbonate, from 30 to 70 wt% of the inorganic salt (preferably sodium carbonate), and from 0.5 to 5 wt% of the polymeric binder.

The total amount of sodium percarbonate in the tabletted composition as a whole is preferably within the range of from 5 to 40 wt%, more preferably from 10 to 30 wt%.

Other ingredients

As well as sodium percarbonate, the detergent tablet of the invention contains at least one detergent-active compound, at least one detergency builder, and optionally other ingredients. Preferred tablets of the invention provide a fully formulated, high performance detergent composition within a single tablet.

Detergent-active compounds

Detergent-active compounds are suitably present in an amount of from 2 to 50 wt%, more preferably from 5 to 40 wt%. Detergent-active material present may be anionic (soap or non-soap), cationic, zwitterionic, amphoteric, nonionic, or any combination of these.

Anionic detergent-active compounds may be present in an amount of from 2 to 40 wt%, preferably from 4 to 30 wt%.

Synthetic anionic surfactants are well known to those skilled in the art. Examples include alkylbenzene sulphonates, particularly sodium linear alkylbenzene sulphonates having an alkyl chain length of C_8-C_{15}; primary and secondary alkyl sulphates, particularly sodium C_{12}-C_{15} primary alcohol sulphates; olefin sulphonates; alkane sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates.

It may also be desirable to include one or more soaps of fatty acids. These are preferably sodium soaps derived from naturally occurring fatty acids, for example, the fatty acids from coconut oil, beef tallow, sunflower or hardened rapeseed oil.

Anionic surfactants are preferably concentrated in discrete domains as described and claimed in our copending application GB 90 15504.5 (Unilever PLC).

Suitable nonionic detergent compounds which may be used include in particular the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example, aliphatic alcohols, acids, amides or alkyl phenols with alkylene oxides, especially ethylene oxide either alone or with propylene oxide.

Specific nonionic detergent compounds are alkyl (C_{6-22}) phenol-ethylene oxide condensates, the condensation products of linear or branched aliphatic C_{8-20} primary or secondary alcohols with ethylene oxide, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylenediamine. Other so-called nonionic detergent compounds include long-chain tertiary amine oxides, tertiary phosphine oxides, and dialkyl sulphoxides.

Especially preferred are the primary and secondary alcohol ethoxylates, especially the C_{12-15} primary and secondary alcohols ethoxylated with an average of from 5 to 20 moles of ethylene oxide per mole of alcohol.

The nonionic detergent-active compounds are preferably concentrated in discrete domains. Since the nonionic detergent compounds are generally liquids, these domains are preferably formed from any of the well-known carriers in the detergent business impregnated by nonionic detergent-active compound. Preferred carriers include zeolite; zeolite granulated with other materials, for example, Wessalith CS (Trade Mark), Wessalith CD (Trade Mark), Vegabond GB (Trade Mark), sodium perborate monohydrate; Burkeite (spray-dried sodium carbonate and sodium sulphate as disclosed in EP 221 776 (Unilever)).

Nonionic detergent-active compounds may optionally be mixed with materials which make the granules slow wetting and/or prevent the nonionic leaching out into the main tablet matrix. Such materials may suitably be fatty acids, especially lauric acid.

Detergency builders

The detergent tablets of the invention contain one or more detergency builders, suitably in an amount of from 5 to 80 wt%, preferably from 20 to 80 wt%.

The invention is of especial relevance to tablets derived from detergent compositions containing alkali metal aluminosilicates as builders, since these builders have a particular tendency to destabilise sodium percarbonate. In the tablet of the invention, aluminosilicate builders must always be excluded from the region containing the sodium percarbonate.

Alkali metal (preferably sodium) aluminosilicates may suitably be incorporated in amounts of from 5 to 60% by weight (anhydrous basis) of the composition, and may be either crystalline or amorphous or mixtures thereof, having the general formula:

$$0.8\text{-}1.5\ Na_2O.\ Al_2O_3.0.8\text{-}6\ SiO_2$$

These materials contain some bound water and are required to have a calcium ion exchange capacity of at least 50 mg CaO/g. The preferred sodium aluminosilicates contain 1.5-3.5 SiO_2 units (in the formula above). Both the amorphous and the crystalline materials can be prepared readily by reaction between sodium silicate and sodium aluminate, as amply described in the literature.

Suitable crystalline sodium aluminosilicate ion-exchange detergency builders are described, for example, in GB 1 429 143 (Procter & Gamble). The preferred sodium aluminosilicates of this type are the well-known commercially available zeolites A and X, and mixtures thereof. Also of interest is the novel zeolite P described and claimed in EP 384 070A.

Other builders may also be included in the detergent tablet of the invention if necessary or desired: suitable organic or inorganic water-soluble or water-insoluble builders will readily suggest themselves to the skilled detergent formulator. Inorganic builders that may be present include alkali metal (generally sodium) carbonate; while organic builders include polycarboxylate polymers such as polyacrylates, acrylic/maleic copolymers, and acrylic phosphinates; monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono-, di- and trisuccinates, carboxymethyloxysuccinates, carboxymethyloxymalonates, dipicolinates, hyd-

roxyethyliminodiacetates; and organic precipitant builders such as alkyl- and alkenylmalonates and succinates, and sulphonated fatty acid salts.

Especially preferred supplementary builders are polycarboxylate polymers, more especially polyacrylates and acrylic/maleic copolymers, suitably used in amounts of from 0.5 to 15 wt%, especially from 1 to 10 wt%; and monomeric polycarboxylates, more especially citric acid and its salts, suitably used in amounts of from 3 to 20 wt%, more preferably from 5 to 15 wt%. As previously indicated, at least part of any polymer required in the formulation may be incorporated, as binder, in the region of the tablet in which the sodium percarbonate is segregated.

Preferred tabletted compositions of the invention preferably do not contain more than 5 wt% of inorganic phosphate builders, and are desirably substantially free of phosphate builders. However, phosphate-built tabletted compositions are also within the scope of the invention.

Bleach activators

To enhance the low-temperature performance of sodium percarbonate, a bleach activator (bleach precursor) is advantageously present. The bleach activator should be excluded from the region in which the sodium percarbonate is segregated.

Preferred bleach activators include peracetic acid precursors, for example, tetraacetylethylenediamine (TAED), and perbenzoic acid precursors. The novel quaternary ammonium and phosphonium bleach activators disclosed in US 4 751 015 and US 4 818 426 (Lever Brothers Company) and the monosaccharide esters as disclosed in EP 0 380 437A (Procter & Gamble; Novo) are also of great interest.

Bleach activators are suitably present in an amount of from 1 to 10 wt%, more preferably from 2 to 5 wt%.

Detergent tablets containing persalts and bleach activators are also described and claimed in our copending British patent application of even date (Case C3390).

Bleach stabilisers

The bleach system may also include a small amount of a bleach stabiliser (heavy metal sequestrant) such as ethylenediamine tetraacetate (EDTA), ethylenediamine tetramethylene phosphonate (EDTMP) or diethylenetriamine pentamethylene phosphonate (DTPMP).

Enzymes

The detergent tablets of the invention may also contain one of the detergency enzymes well-known in the art for their ability to degrade and aid in the removal of various soils and stains. Most enzymes are bleach-sensitive to some extent, and should also be excluded from the region containing the sodium percarbonate.

Suitable enzymes include the various proteases, cellulases, lipases, amylases, and mixtures thereof, which are designed to remove a variety of soils and stains from fabrics. Examples of suitable proteases are Maxatase (Trade Mark), as supplied by Gist-Brocades N.V., Delft, Holland, and Alcalase (Trade Mark), Esperase (Trade Mark) and Savinase (Trade-Mark), as supplied by Novo Industri A/S, Copenhagen, Denmark. Detergency enzymes are commonly employed in the form of granules or marumes, optionally with a protective coating, in amounts of from about 0.1% to about 3.0% by weight of the composition; and these granules or marumes present no problems with respect to compaction to form a tablet.

Minor ingredients

The detergent tablets of the invention may also contain a fluorescer (optical brightener), for example, Tinopal (Trade Mark) DMS or Tinopal CBS available from Ciba-Geigy AG, Basel, Switzerland. Tinopal DMS is disodium 4,4'bis-(2-morpholino-4-anilino-s-triazin-6-ylamino) stilbene disulphonate; and Tinopal CBS is disodium 2,2'- bis-(phenyl-styryl) disulphonate.

An antifoam material is advantageously included in the detergent tablet of the invention, especially if the tablet is primarily intended for use in front-loading drum-type automatic washing machines. Suitable antifoam materials are usually in granular form, such as those described in EP 266 863A (Unilever). Such antifoam granules typically comprise a mixture of silicone oil, petroleum jelly, hydrophobic silica and alkyl phosphate as antifoam active material, sorbed onto a porous absorbent water-soluble carbonate-based inorganic carrier material. Antifoam granules may be present in any amount up to 5% by weight of the composition.

It may also be desirable to include in the detergent tablet of the invention an amount of an alkali metal silicate, particularly sodium ortho-, meta- or preferably neutral or alkaline silicate. The presence of such alkali metal

silicates at levels, for example, of 0.1 to 10 wt%, may be advantageous in providing protection against the corrosion of metal parts in washing machines, besides providing some measure of building and giving processing benefits.

Further ingredients which can optionally be employed in the detergent tablet of the invention include anti-redeposition agents such as sodium carboxymethylcellulose, straight-chain polyvinyl pyrrolidone and the cellulose ethers such as methyl cellulose and ethyl hydroxyethyl cellulose; fabric-softening agents; heavy metal sequestrants such as EDTA; perfumes; pigments, colourants or coloured speckles; and inorganic salts such as sodium and magnesium sulphate. Sodium sulphate may if desired be present as a filler material in amounts up to 40% by weight of the composition; however as little as 10% or less by weight of the composition of sodium sulphate, or even none at all, may be present.

As well as the functional detergent ingredients listed above, there may be present various ingredients specifically to aid tabletting or to aid tablet dispersion in the wash, for example, binders, disintegrants, or lubricants. As already indicated, some ingredients may give both functional wash benefits and tabletting benefits.

Tabletting

As previously indicated, the tablets of the invention are prepared by compaction of particulate starting material. Any suitable compaction process may be used, for example, tabletting, briquetting or extrusion, but tabletting is the preferred and most suitable process.

For any given starting composition, the time taken for the tablet to disintegrate in the wash liquor will vary with the compaction pressure used to form the tablet. If the compaction pressure is too low, the tablet will tend to crumble and break up in the dry state, on handling and packaging; an increase in compaction pressure will improve tablet integrity, but eventually at the expense of disintegration time in the wash liquor.

Using an Instron (Trade Mark) Universal Testing Machine at constant speed, or a Research and Industrial screw hand press, to operate a steel punch and die, it has been found that effective tablets may be produced using compaction pressures ranging from 0.1 to 500 MPa (0.01 to 50 kN/cm^2), especially from 0.2 to 100 MPa (0.02 to 10 kN/cm^2).

The optimum compaction pressure will depend to some extent on the starting composition; for example, a formulation containing a high proportion of organic ingredients (for example, surfactants) and a low proportion of inorganic salts may require a compaction pressure lower than that required for a formulation containing a lower proportion of organic ingredients and a higher proportion of inorganic salts; and a dry-mixed formulation will generally require a higher pressure than will a spray-dried powder.

Preferred tablet forms

Preferred tablets having improved disintegration and dissolution properties are described and claimed in our copending British Patent Applications Nos. 90 15503.7 and 90 15504.5 (Unilever PLC) filed on 13 July 1990, and our copending British Patent Application filed on 1 July 1991 (case C3408) (Unilever PLC). These preferred tablet forms have particular relevance for tablets of fully formulated detergent compositions.

The tablet described and claimed in Application No. 90 15503.7 or a discrete region thereof, consists essentially of a matrix of particles substantially all of which have a particle size within a range having upper and lower limits each lying within the range of from 200 to 2000 μm and differing from each other by not more than 700 μm.

According to Application No. 90 15504.5, a tablet of compacted particulate detergent composition comprises a minor proportion (2-40 wt%) of a first component (a) which contains 20-100 wt% anionic surfactant, the rest of the composition containing only 0-3 wt% anionic surfactant.

The tablet described and claimed in our copending British Application C3408, filed on 1 July 1991, or a discrete region thereof, consists essentially of a matrix of particles substantially all of which have a particle size >200μm, at least the particles of detergent-active compound and detergent builder are coated with binder/disintegrant before tablet compaction.

Dosage forms

The detergent tablet of the invention may be, and preferably is, formulated for use as a complete heavy-duty fabric washing composition. The consumer does not need to use a mix of tablets having different compositions.

Although one tablet may contain sufficient of all the components to provide the correct amount required for an average washload, it is convenient if each tablet contains a submultiple quantity of the composition required for average washing conditions, so that the consumer may vary the dosage according to the size and

nature of the washload. For example, tablet sizes may be chosen such that two tablets are sufficient for an average washload; one or more further tablets may be added if the washload is particularly large or soiled; and one only tablet may be used if the load is small or only lightly soiled.

Alternatively, larger subdivisible tablets representing a single or multiple dose may be provided with scorings or indentations to indicate unit dose or submultiple unit dose size to the consumer and to provide a weak point to assist the consumer in breaking the tablet if appropriate.

The size of the tablet will suitably range from 10 to 160 g, preferably from 15 to 60 g, depending on the wash conditions under which it is intended to be used, and whether it represents a single dose, a multiple dose or a submultiple dose.

The tablet may be of any suitable shape, but for manufacturing and packaging convenience is preferably of uniform cross-section, for example, circular (preferred) or rectangular.

EXAMPLES

The following non-limiting Examples illustrate the invention. Parts and percentages are by weight unless otherwise stated. Examples identified by numbers are win accordance with the invention, while Examples identified by letters are comparative.

Example 1, Comparative Example A

(i) Preparation of bleach composition

A 40 wt% solution of Analar sodium carbonate was prepared. Acrylic/maleic copolymer in sodium salt form - sokalan (Trade Mark) CP5 ex BASF - was admixed in an amount of 2 wt% based on the sodium carbonate (dry weight), and the solution was stirred at 50°C for 2 hours. The solution was then spray-dried using laboratory equipment (inlet temperature 275°C, feed rate 10 ml/min through a 0.75 mm jet) to give granular anhydrous sodium carbonate of high specific surface area.

A bleach composition was then prepared by dry-mixing equal weights of the spray-dried sodium carbonate composition and sodium percarbonate.

(ii) Preparation of detergent base composition

A detergent base composition was prepared to the formulation shown in Table 1, by spray-drying an aqueous slurry of all ingredients except the nonionic surfactant 3EO which was subsequently sprayed on.

(ii) Tabletting

Tablets were prepared using an Instron (Trade Mark) Model 4202 Materials Testing Machine fitted with a 10kN load cell. The machine was operated at its maximum speed of 100 mm/min and at a pressure of 0.14 kN/cm^2 (1.4 MPa).

For Example 1 (the invention), bleach composition (10 g) was added to the die, the die was tapped gently to level the powder, and detergent base composition (30 g) was added on top of the bleach composition, before tabletting.

For Comparative Example A, the same weights of bleach composition and detergent composition as in Example 1 were mixed together, and the mixture added to the die for tabletting.

The tablets each weighed 40 g, and were 53 mm in diameter and 22 mm in thickness.

(iii) Storage

The tablets were stored under two sets of conditions: at 37°C/70% relative humidity, and at 28°C/70% relative humidity. The storage regime was deliberately chosen to be as adverse as possible: the tablets, totally unprotected by any packaging, were placed standing on edge and separated from one another so as to expose the maximum possible surface area to the atmosphere.

(iv) Dissolution tests

Dissolution testing was carried out in a Miele (Trade Mark) 756 front-loading automatic washing machine, using the 40°C wash cycle without a washload:

dissolution was monitored using a conductivity cell. The results are given as the times taken, to the nearest minute, for 50 wt% and 90 wt% dissolution to take place. The results are shown in Table 2.

Table 1: Detergent Base and Tablet Compositions

	parts	% (tablet)
Detergent base composition		
Linear alkylbenzene sulphonate	6.00	7.16
Nonionic surfactant (7EO)	2.75	3.28
Soap	1.65	1.97
Zeolite 4A (anhydrous basis)	24.00	28.64
Polymer (acrylic/maleic)	4.00	4.77
Sodium alkaline silicate	0.46	0.55
Sodium carbonate	8.13	9.70
Sodium carboxymethylcellulose	0.50	0.60
Fluorescer	0.19	0.23
EDTA	0.20	0.24
Salts, moisture	10.72	12.79
Nonionic surfactant 3EO	4.25	5.07
	62.85	75.00
Tablet Composition		
Detergent base composition	30.0	
Sodium percarbonate	5.0	12.5
Spray-dried sodium carbonate	5.0	12.5
	40.00	100.0

Table 2: Dissolution Results

(i) 28°C/70% relative humidity

Storage time (weeks)	Example 1		Example A	
	50%	90%	50%	90%
1	2	9	10	30
2	2	9	15	25
4	3	12	20	>30
6	3	17	25	>30

(ii) 37°C/70% relative humidity

Storage time (weeks)	Example 1		Example A	
	50%	90%	50%	90%
2	2	12	8	23
4	4	14	8	27
6	Tablets disintegrated during storage			

(v) Bleach stability

Percarbonate storage stability was determined by available oxygen titration using potassium permanganate, the results being expressed as residual available oxygen as a molar percentage of the theoretical value. The results are shown in Table 3.

Table 3: Percarbonate storage stability

(i) 28°C/70% relative humidity

Storage time (weeks)	Remaining available oxygen (mole %)	
	Example 1	Example A
0	100	100
1	91	59
2	83	22
4	70	2
6	68	0
8	63	0

(ii) 37°C/70% relative humidity

Storage time (weeks)	Remaining available oxygen (mole %)	
	Example 1	Example A
0	100	100
2	75	20
4	50	0
6	48	0

Examples 2 and 3

(i) Preparation of bleach composition

Example 2

Burkeite was made by spray-drying a mixture of sodium carbonate, sodium sulphate and Sokolan (Trade Mark) CP5 as described in EP 221776 (Unilever).

A bleach composition was then prepared by dry-mixing equal weights of the spray-dried Burkeite compo-

sition and sodium percarbonate.

Example 3

Spray dried sodium carbonate/Sokolan (Trade Mark) CP5 was prepared as in Example 1.

A bleach composition was then prepared by dry-mixing equal weights of the spray-dried sodium carbonate composition and sodium percarbonate.

(ii) Preparation of detergent base composition

A detergent base composition was prepared to the formulation shown in Table 4, by spray-drying on aqueous slurry of all the ingredients except the nonionic surfactant 3EO which was subsequently sprayed on and the PAS noodles which were posed-dosed.

(iii) Tabletting

As for Example 1.

(iv) Storage

As for Example 1.

Table 4. Detergent Base and Tablet Composition

	Parts	% (tablet)
Detergent base composition		
Nonionic surfactant (7EO)	4.5	3.95
Zeolite 4A	37.0	32.45
Polymer (acrylic/maleic)	5.0	4.39
Sodium carbonate	14.9	13.07
Sodium carboxymethyl cellulose	0.5	0.44
Fluorescer	0.2	0.18
Moisture	11.0	9.65
Nonionic surfactant (3EO)	4.0	3.50
PAS Noodles	8.4	7.37
	85.5	75.00
Tablet Composition		
Detergent Base Composition	30.0	
Sodium percarbonate	5.0	12.5
Spray-dried Burkeite (Example 2) or Sodium carbonate (Example 3)	5.0	12.5
	40	100

(v) Bleach Stability

Percarbonate storage stability was determined as in Example 1.
The results are shown in Table 5.

Table 5. Percarbonate Storage Stability

(i) 28°C/70% relative humidity

Storage time (Weeks)	Remaining available oxygen (mole %)	
	Example 2	Example 3
0	100	100
2	70	83
4	71	-
5	60	50

(ii) 37°C/70% relative humidity

Storage time (Weeks)	Remaining available oxygen (mole %)	
	Example 2	Example 3
0	100	100
2	71	77
4	72	38
5	60	23

Claims

1 A tablet of compressed particulate detergent composition comprising a detergent-active compound, a detergency builder, a bleach system comprising sodium percarbonate, and optionally other detergent ingredients, characterised in that the percarbonate is separated from any ingredient of the composition detrimental to its stability by segregation in a discrete region of the tablet.

2 A tablet as claimed in claim 1, characterised in that the discrete region contains sodium percarbonate and a compatible inorganic salt.

3 A tablet as claimed in claim 2, characterised in that the discrete region contains sodium percarbonate and a spray-dried diluent comprising a compatible inorganic salt and a polymeric binder.

4 A tablet as claimed in claim 2 or claim 3, characterised in that the compatible inorganic salt comprises sodium carbonate.

5 A tablet as claimed in any one of claims 1 to 4, characterised in that the detergency builder comprises alkali metal aluminosilicate and is excluded from the discrete region.

6 A tablet as claimed in any one of claims 1 to 5, characterised in that a bleach activator is present and is excluded from the discrete region.

7 A tablet as claimed in any one of claims 1 to 6, characterised in that the discrete region is in the form of a layer, a core or an insert.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 91 30 9598

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
A	FR-A-2 236 930 (HENKEL & CIE GMBH) * page 2, lines 10-21; page 3; page 4, lines 1-18; page 8, lines 16-21 *	1	C 11 D 17/00 C 11 D 3/39
A	FR-A-2 249 164 (KAO SOAP CO) * claims *; & US - A - 3953350 (Cat. D)	1	
A	DE-A-2 622 610 (INTEROX BRÜSSEL) * claims *	1	
D,A	GB-A- 911 204 (UNILEVER LTD) * claims *	1	

TECHNICAL FIELDS SEARCHED (Int. Cl.5)

C 11 D

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
BERLIN	16-01-1992	PELLI-WABLAT B

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

& : member of the same patent family, corresponding document

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